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MULTIFUNCTIONAL EXPLOSIVE BATTERY (MEB) STUDY

LOCKHEED MISSILES & SPACE COMPANY

TECHNICAL REPORT AFATL-TR-71-36

MARCH 1971

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Multifunctional Explosive Battery (MEB) Study

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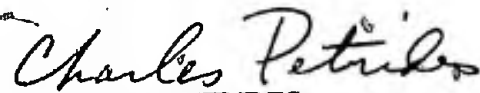
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FOREWORD

This report documents work accomplished during the period July 1970 through January 1971 by the Advanced Concepts Laboratory of the Lockheed Palo Alto Research Laboratory, Lockheed Missiles & Space Company, Palo Alto, California, under Air Force Contract F08635-70-C-0115, entitled "Multifunctional Explosive Battery Study." This work was sponsored by the Air Force Armament Laboratory, Eglin Air Force Base, Florida, and the program was monitored by Mr. William D. Creamer, Captain Donald D. Potter, and Captain Michael P. Culley (DLDF), of the Armament Laboratory.

Lockheed Missiles & Space Company personnel chiefly responsible for the performance of this work were Mr. F. F. Stucki and Mr. H. F. Bauman, Staff Scientists, Dr. J. E. Chilton, Research Scientist, and Mr. J. Troendle, Staff Engineer.

This report has been reviewed and is approved.



CHARLES PETRIDES

Acting Chief, Advanced Development Division

ABSTRACT

The feasibility of a multifunctional explosive battery was established by the detonation of metal-cased cells which had previously been discharged to supply electrical power. These batteries were based on the lithium-copper hydroxyfluoride electrochemical system with an electrolyte of nitromethane, ethylene carbonate, lithium perchlorate, and aluminum chloride. The electrolyte was the explosive component in the cell and comprised less than 25 percent of the cell weight. A problem of limited compatibility exists between the lithium anode and the combination of nitromethane and lithium perchlorate in the electrolyte. Sufficient stability to complete testing was obtained by proper selection of electrolyte additives.

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SECTION I INTRODUCTION

The purpose of this program was to explore the feasibility of adding additional functions to the battery used to power target activated munitions. Primary interest, besides supplying power, was in using cell components that could be detonated either to augment the explosive charge of the munition or as an explosive booster in the firing train. Other functions that were considered include timing, programing, level sensing, and disturbance sensing. The use of a multifunctional battery can simplify construction and assembly, may serve as an additional safety device, and will increase the effectivity of a munition without an increase in weight or size.

Detonation-sensitive electrolytes may be prepared from organic solvents that dissolve and ionize inorganic salts. These solvents may be selected to have a wider liquid range than water, allowing (1) a greater temperature range for operation as well as a much higher decomposition potential than with water, and (2) use of more active anodic materials (such as the alkali metals) in a cell. Such cells would have a higher energy density than cells with aqueous electrolytes. Since the electrolyte is a significant portion of the weight of a cell, it is a reasonable choice as the explosive component. Cathodic materials with explosive capability (e. g., dinitrobenzene) could also be considered, but their explosive power would diminish during discharge when reduced compounds formed as the result of electrochemical energy production.

The program was organized in two phases: (1) a literature study to select an electrochemical system to establish feasibility, and (2) fabrication and testing of cells based on the system selected.

SECTION II SYSTEM SELECTION

2.1. SELECTION CRITERIA AND CANDIDATE MATERIALS

The major criteria in the selection of components are that they deliver electrical power when combined in a battery and that one or more are explosive but will operate safely over a range of temperatures and physical operating conditions. The electrical requirements used in the selection are 20 milliamperes of current delivered for 15 days at 3 volts with no surges. If an electrode system with a potential of 3 volts is selected, a single 7.2-ampere-hour cell will meet the requirement. The cell must have sufficient stability to operate for 15 days with as wide a temperature range as possible, or approaching the -65 to $+165^{\circ}\text{F}$ range. The materials of the battery must be chosen so that, with proper mechanical construction, they will tolerate shock and vibration.

2.2 SELECTION OF ELECTRODE MATERIALS

Materials considered for the cell anode were sodium and lithium. The physical properties and experimentally obtained electrochemical values are shown in Table I.

Although polarization and utilization data for sodium were not reported in the literature studied, experimental values obtained at the contractor's facility were available. Lithium had several advantages over sodium as a cell anode for this application.

- Its utilization and polarization appeared to be tolerable for the intended development.
- It has a higher theoretical energy density than sodium and, equally important, a higher electrode potential; this had been demonstrated experimentally.
- Recent work at contractor's facility (substantiated by work reported by another laboratory) indicated that sodium attacks solvents, such as propylene carbonate and butyrolactone, which are stable with lithium.

Economically, sodium costs approximately one-tenth as much as lithium for a given ampere-hour of discharge capacity; however, since the cost of lithium is calculated at less than half a cent per ampere-hour, its use in low-cost items does not appear to be precluded.

Materials considered for the cell cathode were cupric fluoride (CuF_2), cupric chloride (CuCl_2), cupric sulfide (CuS), and silver chloride (AgCl). The physical properties of these materials and the reported performance figures are shown in Table II. The cathode data were obtained from reported cell discharges and were affected by the electrolyte and the counterelectrode. The figures were not directly comparable because the cells were of different mechanical structure and were assembled and discharged in different laboratories. The cathode structure and the electrode spacing, which have a large effect on the cell potential during discharge, were undoubtedly different in the several cases.

TABLE I. COMPARISON OF SODIUM (Na) AND LITHIUM (Li) AS ANODES

Parameter	Na	Li
Molecular Weight	22.99	6.94
Density	0.97	0.53
Melting Point (°C)	97.5	186
Potential Versus H ₂ (Acetonitrile) (V)	2.87	3.23
Methyl Formate	2.81	3.12
Coulombic Capacity (Ampere-Hour per Pound)	529	1752
Current Density Achieved Experimentally	10 mA/in. ² (< 1 hr)	10 mA/in. ² (100 hr)
Utilization Achieved Experimentally	30%	60-85%
Reactivity	Reacts with propylene carbonate and butyrolactone	Stable nitromethane and propylene carbonate
Cost (Cents per Ampere- Hour)	0.038	0.428

The cathode constitutes the major portion of the weight of the cell reactants, as can be seen by comparing the ampere-hours per pound using sodium or lithium. On this basis, CuS has the best theoretical ampere-hour figure, and excellent discharges are realized in practice. Specific solvents may be necessary to achieve such results, however. In addition, the cell potential is low and, for the present application, two cells would be required to obtain the required potential during discharge.

Cupric fluoride has the best watt-hours per pound figure of the cathodes listed because of its higher cell potential. With lithium as the anode, it has an open-circuit potential voltage of 3.45 volts per cell and discharges have been obtained at over 3 volts. Lithium-cupric fluoride cells in welded aluminum cases have delivered in excess of 80 watt-hours per pound and, when placed in plastic cases, over 100 watt-hours per pound.

Cupric chloride has been used in cells designed for high discharge rates and has given reasonable efficiency and discharge voltage. Its voltage is lower than that of CuF₂ and, because the higher rate is not necessary in this application, the higher energy content of the fluoride may make it the best choice. CuCl₂ is quite soluble in many solvents, which might prevent attainment of the desired discharge life.

TABLE II. COMPARISON OF CATHODE MATERIALS

Material	Molecular Weight	Density	Melting Point (°C)	Nonaqueous Cell Potential Versus Li (volts)	Coulomb Capacity (ampere-hour per pound)	Experimental Current Density Achieved (milliamperes per square inch)	Utilization (percent)	Theoretical Energy Density of Couple With Li Anode (watt-hours per pound)	Experimental Energy Density Achieved With Li Anode Cells (watt-hours per pound)	Cost (cents per ampere-hour)
Cupric Fluoride (CuF ₂)	101.54	4.23	Decomposed at 958	3.45	239	1.5 (200 + hr)	70	746	109	1.2
Cupric Chloride (CuCl ₂)	134.48	3.05	498	3.42	181	68 (1.0 hr)	48	502	109	0.88
Cupric Sulfide (CuS)	95.63	4.6	Decomposed at 220	2.15	254	1.5 (250 hr)	Good	488	127	0.89
Silver Chloride (AgCl)	143.34	5.56	455	2.90	85	5.0 (1.5 hr)	75-90	229	20	11.4

Silver chloride has reasonable electrochemical characteristics in use, but its weight lowers the energy density of any cell of which it is a component. In addition, it is the most expensive of the cathodic material being considered.

In selecting components for a battery system where only electrical power is required, the electrodes and electrolyte are usually selected on the basis of material compatibility and the required discharge profile. In this case, however, the electrolyte must be explosive as well as being a conductive and compatible component of the battery. This limits the solvents that can be used, since only a few liquids are considered safe explosives or have been used as explosives. The characteristics of the solvents considered are shown in Table III. The first four solvents are explosive; Propylene Carbonate (PC) and Ethylene Carbonate (EC) are not explosive and are to be used only as diluents to alter the electrolyte properties.

The calculated explosive power of a compound is optimized when all the products are oxidized gases or N_2 , which in the case of common explosives would be CO , H_2O , and N_2 . On this basis, the solution of oxidizing salts (perchlorates or nitrates) may be used to increase the oxygen present in the electrolyte for reaction. It may then be feasible to use liquids which themselves are nonexplosive to increase conductivity or to change the melting point and vapor pressure of the electrolyte.

Except for nitromethane, only limited electrochemical data were available for the other liquids included as explosive. In one report, a statement was made that 1- and 2-nitropropane were poor solvents for salts and their use was discontinued. Another report states that $LiClO_4$ was not sufficiently soluble in methyl nitrate to make a 0.1-M solution.

Nitromethane has been used by itself and with propylene carbonate as a solvent for $LiAlCl_4$ and $AlCl_3$ in cell systems using $Li-AgCl$ and $Li-CuCl_2$ electrode couples. It is compatible with lithium, and its chloride and sodium hexafluorophosphate solutions have better than average conductivity for nonaqueous systems (greater than $10^{-2} \Omega^{-1} cm^{-1}$). It was hoped that ethylene carbonate, with its higher dielectric constant, could be used as an additive both to increase the conductivity of the electrolyte and to reduce the vapor pressure.

A ratio of the energy quantity characteristic of an explosive may be calculated and compared with a standard such as trinitrotoluene (TNT) by the use of thermochemical data to determine values of the perfect gas expression nRT . These ratios have been correlated with Trauzl lead block tests, which is an empirical method of obtaining explosive strength ratios. The reactions are written with N_2 , CO , H_2O , and H_2 as products and the heat of reaction H_r calculated from the heats of formation of the reactants and the products. From prepared tables of average specific heats for the product substances between $300^\circ K$ and various upper limit temperatures, the adiabatic explosion temperature T is obtained by a trial-and-error procedure. The nT product is calculated on a molar basis and compared with that of TNT. A similar gas constant is assumed for the reactions and is eliminated from the calculations when the data are presented as a ratio. Table IV presents the results of calculations of some of the materials with and without added lithium perchlorate. The amount of $LiClO_4$ necessary to obtain a zero oxygen balance with nitromethane corresponds to an 18 percent solution by weight. These values give some insight into explosive forces; however, actual products may differ from those used in the calculations and some of the approximations may be additive, so that, in the

TABLE III. PROPERTIES OF NONAQUEOUS SOLVENTS

Solvent	Structure	Molecular Weight	Melting Point (°C)	Boiling Point (°C)	Density (g/cm ³ at 25°C)	Dielectric Constant at 30°C	Viscosity Cp 10°C/75°C	Conductivity (Ω ⁻¹ cm ⁻¹)			Explosive
								1.0M LiClO ₄	1.0M NaPF ₆	1.0M LiAlCl ₄	
Nitro-Methane	<chem>C[N+](=O)[O-]</chem>	61	-28.5	101.2	1.131	35.89	0.731/ 0.610	-	1.2×10^{-2} (sat.)	4.2×10^{-2} (1.4M)	Yes
1-Nitro-Propane	<chem>CCCN=[N+]=[O-]</chem>	89	-104	131.2	0.996	23.24	0.871/ 0.790	-	-	-	?
Ethyl Nitrate	<chem>CCOC(=O)N=[N+]=[O-]</chem>	91	-102	88.7	1.105	-	-	-	-	-	Yes
Diethylene Glycol Dinitrate	<chem>OCC(OC(=O)N=[N+]=[O-])OCC(OC(=O)N=[N+]=[O-])O</chem>	196	2	160	1.365	-	- / 8.1	-	-	-	Yes
Propylene Carbonate	<chem>C1CCOC1=O</chem>	102	-49	241	1.19	65 (25°C)	- / 2.53	5.6×10^{-3}	7.0×10^{-3}	-	No
Ethylene Carbonate	<chem>C1CCOC1=O</chem>	88	36	248	-	95 (25°C)	-	-	-	-	No

TABLE IV. CALCULATED PROPERTIES OF EXPLOSIVE LIQUIDS

Reactant(s)	Products	$\frac{W}{M}$ (g/M)	Moles of Gas n	Heat of Reaction ΔH_r (kcal/M)	Tem- perature of Reaction T (°K)	$\frac{nT}{W}$	Ratio $\frac{nRT}{nRT_{TNT}}$
Trinitro Toluene ($C_7H_5N_3O_6$)	$\frac{3}{2} N_2 + 7CO + 2H_2O + \frac{1}{2} H_2$	227	11	-288	4030	195	1.00
Nitromethane (CH_3NO_2)	$\frac{1}{2} N_2 + CO + H_2O + \frac{1}{2} H_2$	61	3	-57.2	3010	148	0.76
Nitromethane + $LiClO_4$ ($CH_3NO_2 + \frac{1}{8} LiClO_4$)	$\frac{1}{2} N_2 + CO + \frac{3}{2} H_2O + \frac{1}{8} LiCl$	74.3	3	-89.1	3900	157	0.81
Nitroethane ($C_2H_5NO_2$)	$\frac{1}{2} N_2 + CO + H_2O + C + \frac{3}{2} H_2$	75	4	-52.2	2020	107.5	0.55
Ethyl Nitrate ($C_2H_5NO_3$)	$\frac{1}{2} N_2 + CO + 2H_2O + C + \frac{1}{2} H_2$	91	4	-96	2980	131	0.67
Nitropropane ($C_3H_7NO_2$)	$\frac{1}{2} N_2 + CO + H_2O + 2C + \frac{5}{2} H_2$	89	5	-44.3	1490	84	0.43

final analysis, experimental results must be obtained, particularly for complex systems involving solvent, solute, separator, and electrodes.

On the basis of the various advantages and disadvantages for the cell electrodes and electrolyte, the electrochemical system selected for feasibility testing, based on the electrical requirement of delivering 20 milliamperes for 15 days at a 3-volt potential, was the lithium-copper fluoride coupled with an electrolyte of predominantly nitromethane-lithium perchlorate.

SECTION III

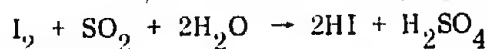
COMPONENT DEFINITION

3.1 ELECTROLYTE

3.1.1 Water Determination

Water is the impurity that has the greatest effect on cell properties. Not only does it affect solubility and conductivity but it reacts with active anode materials. All solvents used in tests were treated with a Linde 4A molecular sieve as a dehydrating agent. This sieve is a highly porous synthetic zeolite that adsorbs molecules of 4 Å or less while excluding molecules of larger diameter. Besides water, it adsorbs H₂S, CO₂, SO₂, and paraffinic hydrocarbons to C₃H₆. It will dry gas to a dewpoint below -120°F and has an equilibrium H₂O capacity of 22 percent at 25°C. For solvent dehydration, the sieve pellets were washed in water to remove soluble salts and were dried in a vacuum at 320°C for 16 hours. The equipment used allowed drying of the molecular sieve in a vacuum and transfer of the material to an inert atmosphere glove box for mixing with the solvent without exposure to air.

Water concentration in solvents and electrolytes was determined with a Karl Fischer titration. The reagent for this titration (Karl Fischer Reagent) is a solution of iodine, sulfur dioxide, and pyridine in methanol. In the presence of sulfur dioxide, iodine reacts with water according to the reaction



The pyridine is necessary in the reagent to prevent a reduction in pH by the acid and reversal of the iodine reduction. The signal of endpoint was obtained in a microammeter by placing a potential across the electrodes (Figure 1) and measuring the current. While water is present and the normal reaction above takes place, polarization of the electrodes occurs and little current flows. The first free iodine from the electrolyte reagent, which does not react because all the water present has reacted, depolarizes the electrodes and the current increases, indicating the endpoint of the titration. Figure 1 also shows the sealed reaction vessel that prevents water from the air from affecting the determination. Values obtained for electrolytes indicated water levels were generally less than 100 ppm.

3.1.2 Freezing Point Determinations

Freezing point determinations were made on selected electrolytes contained in small capped vials. The vials were immersed in ethanol contained in a test tube with a thermometer, and the tube was cooled in an ethanol-dry ice mixture. For these determinations, approximate temperatures for the formation of first crystals and the point

at which there was no liquid remaining were computed. The results of these freezing point determinations are given in Table V.

TABLE V. PROPERTIES OF 2M LiClO_4 - MIXED SOLVENT ELECTROLYTE

Solvent Added to Nitromethane	Volume Percent Nitromethane	H ₂ O Concentration (ppm)	Conductivity 25°C ($\Omega^{-1} \text{cm}^{-1}$)	Conductivity 0°C ($\Omega^{-1} \text{cm}^{-1}$)	All Frozen (°C)	All Liquid (°C)
1-Nitropropane	80	95	8.9×10^{-3}	6.7×10^{-3}	-50	+15
Propylene Carbonate	80	58	7.9×10^{-3}	5.3×10^{-3}	-40	-40
Ethylene Carbonate	80	94	9.4×10^{-3}	6.4×10^{-3}	-55	-40
Butyrolactone	80	48	8.5×10^{-3}	5.7×10^{-3}	-50	+15
Nitroethane	60	53	7.1×10^{-3}	5.4×10^{-3}	-50	+15
Propylene Carbonate	60	52	6.9×10^{-3}	4.1×10^{-3}	-52	-42
Ethylene Carbonate	60	69	8.1×10^{-3}	4.7×10^{-3}	-50	-10

3.1.3 Conductivity Tests

The conductivity of electrolytes was measured in a glass cell capable of being immersed in a constant-temperature bath (Figure 2). The cell was filled in the glove box and sealed, then removed from the glove box and placed in a constant-temperature bath. Resistance was measured with a General Radio Company 1650-A Impedance Bridge operated at 1000 Hz. Resistance values were converted to conductivity values by the relationship

$$\text{Conductivity } (\Omega^{-1} \text{cm}^{-1}) = \frac{K}{R}$$

where R is the resistance value in ohms and K is the conductivity cell constant determined using a standard resistance electrolyte. Values obtained with electrolytes containing 1 M or greater concentration were about $10^{-2} \Omega^{-1} \text{cm}^{-1}$. In cases where little salt was soluble, the conductivity was much less; in these cases, solubility was estimated by the equation

$$\text{Solubility} = \frac{R}{\Lambda}$$

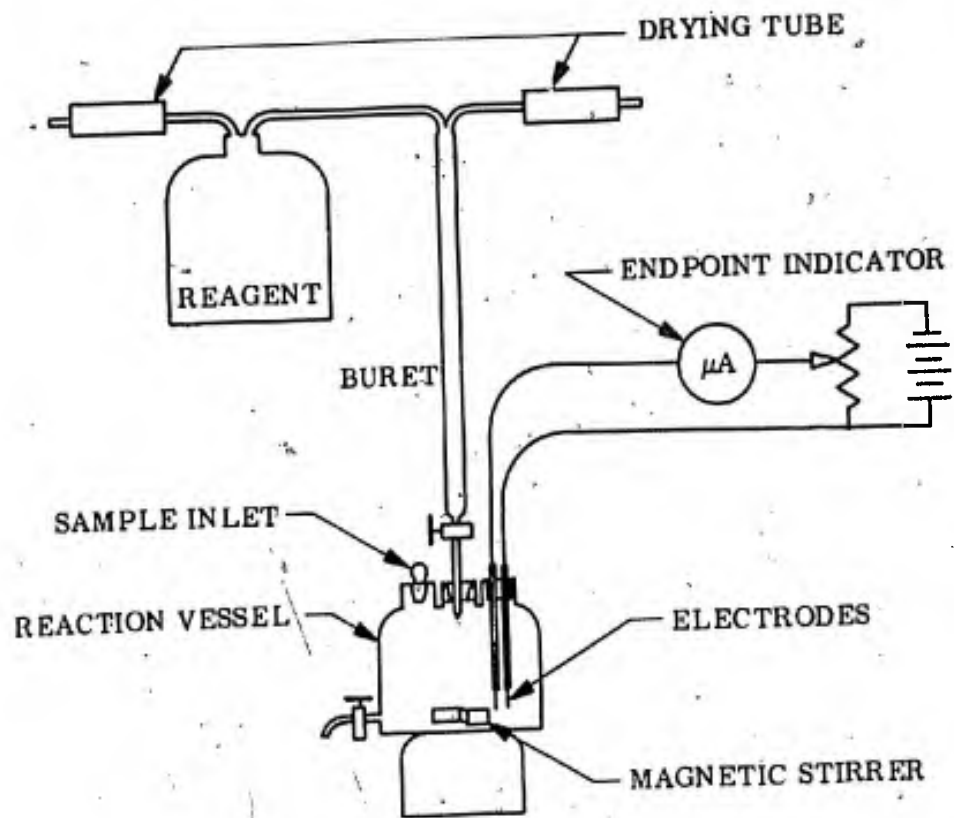


Figure 1. Karl Fischer Titration Apparatus

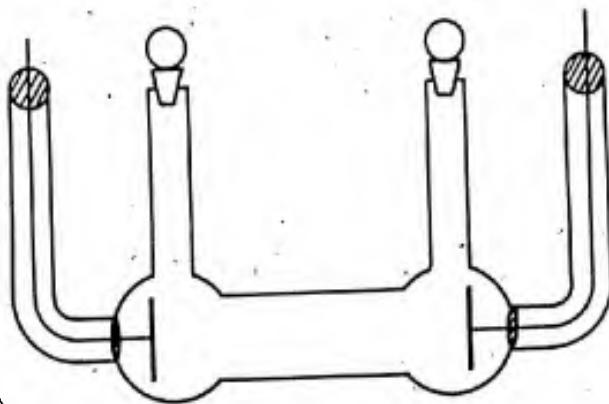


Figure 2. Conductivity Cell

where R is the resistance of the solution and Λ the conductivity measured at infinite dilution. It was found that LiNO_3 was relatively insoluble, and LiClO_4 was used for all electrolyte tests.

A summary of the properties of 2M LiClO_4 , nitromethane, and other solvents is shown in Table V. Propylene carbonate was selected initially as the solvent which, when added to nitromethane, lowered the freezing point most effectively. The conductivity of 80 percent nitromethane-20 percent propylene carbonate solutions of LiClO_4 is shown in Figure 3. The two peaks on this curve may be the result of solvent-salt complexing. Freezing point determinations of solutions with nitromethane also seemed to indicate formation of a solvent-salt precipitate in a 1:1 ratio.

3.1.4 Detonation and Sensitivity Tests

Trauzl or lead block tests were made with selected electrolytes to determine if they could be detonated and to gain knowledge of the explosive power of the composition. The Trauzl tests were made in cylindrical lead blocks eight inches in diameter and eight inches high with a one-inch-diameter, five-inch-deep hole from one face on the axis of the cylinder. The liquid was contained in a glass vial with a cap machined to receive the detonator. All Trauzl tests were conducted with dry sand tamping. The results of these tests, corrected to a 10-gram sample, are shown in Table VI. The Trauzl block, detonator, and sample holder are shown in Figure 4; Figure 5 shows sections of a Trauzl block after a detonator was fired (Test 1) and after 2M LiClO_4 -nitromethane was fired (Test 3).

Some conclusions may be drawn from the Trauzl tests. Nitromethane combined with lithium perchlorate has slightly better explosive properties than the solvent alone. A greater concentration of lithium perchlorate is helpful in obtaining complete combustion of the propylene carbonate. (Smoke was observed after the test with 0.5 M (LiClO_4 .) Ethylene carbonate appears to react more thoroughly than propylene carbonate during the explosion because of its one-less- CH_4 group and requires less oxygen for combustion. Electrolytes may be prepared that have explosive force, as measured by this test, comparable to that of nitromethane and about 25 percent greater than that of TNT.

The sensitivity of an explosive to shock is determined by impact tests in which a weight is dropped on the sample. The drop height that results in a consistent reaction can be used to compare sensitivity. Apparatus designed by personnel at Picatinny Arsenal or the U.S. Bureau of Mines is commonly used for these tests. The apparatus are similar in that several different weights can be used for materials of widely divergent sensitivity but differ in their containment of the sample.

A Bureau of Mines impact tester was used for a sensitivity test of the battery electrolyte. In this test, 0.05 ml of the liquid sample was placed on a 1-cm disc of Whatman No. 1 filter paper, the plunger positioned on the paper, and the weight dropped. The maximum drop height was 150 cm. From this height, a 2-kg weight was dropped on samples of nitromethane, nitromethane-2M LiClO_4 , 80 percent nitromethane-20 percent ethylene carbonate-2M LiClO_4 , and 90 percent nitromethane-10 percent ethylene diamine without any reaction in the sample. The test was repeated with the sample sandwiched between two pieces of sandpaper (to increase sensitivity by the development

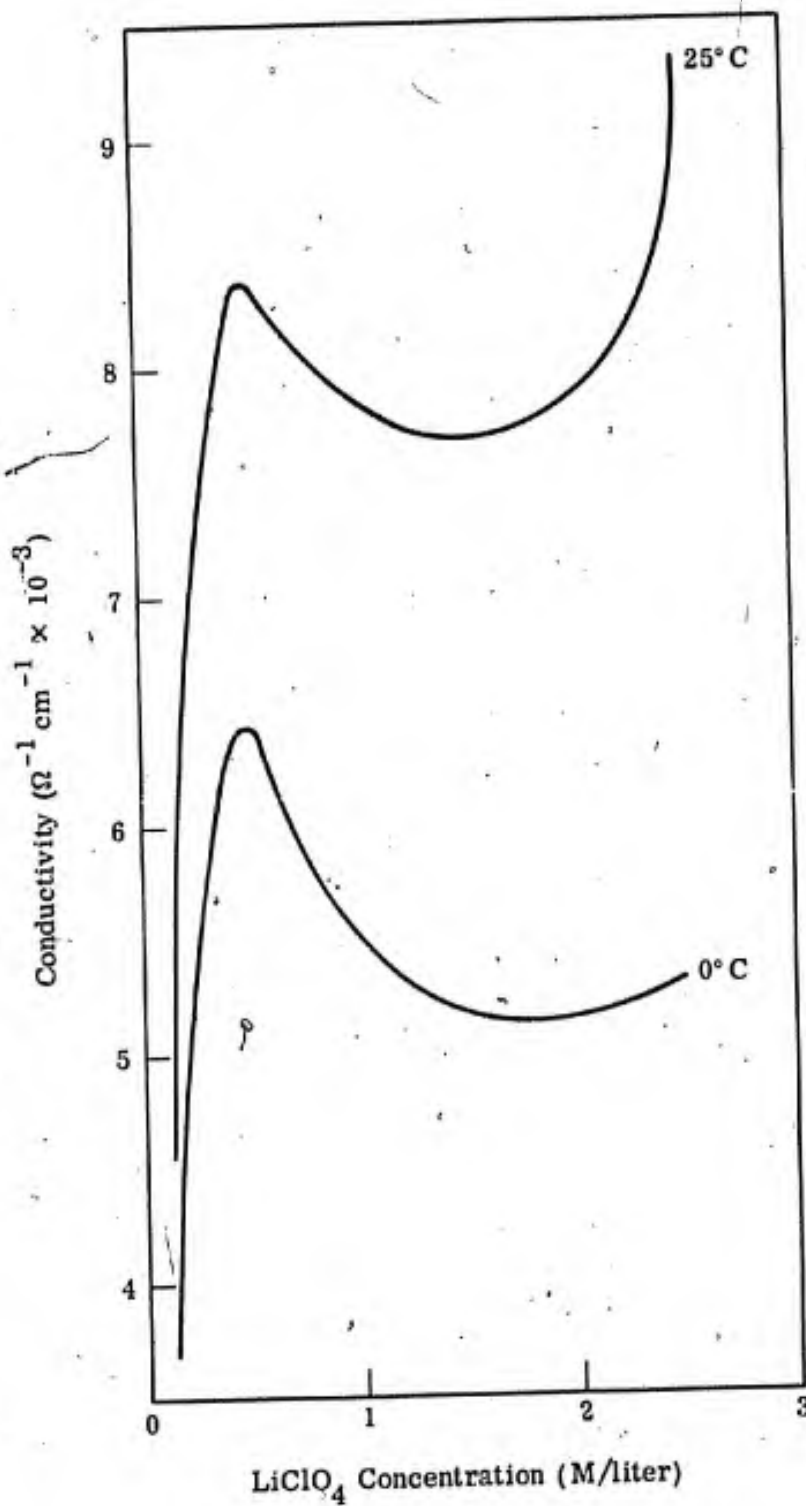


Figure 3. Conductivity of LiClO_4 Solutions in 80 Percent Nitromethane – 20 Percent Propylene Carbonate

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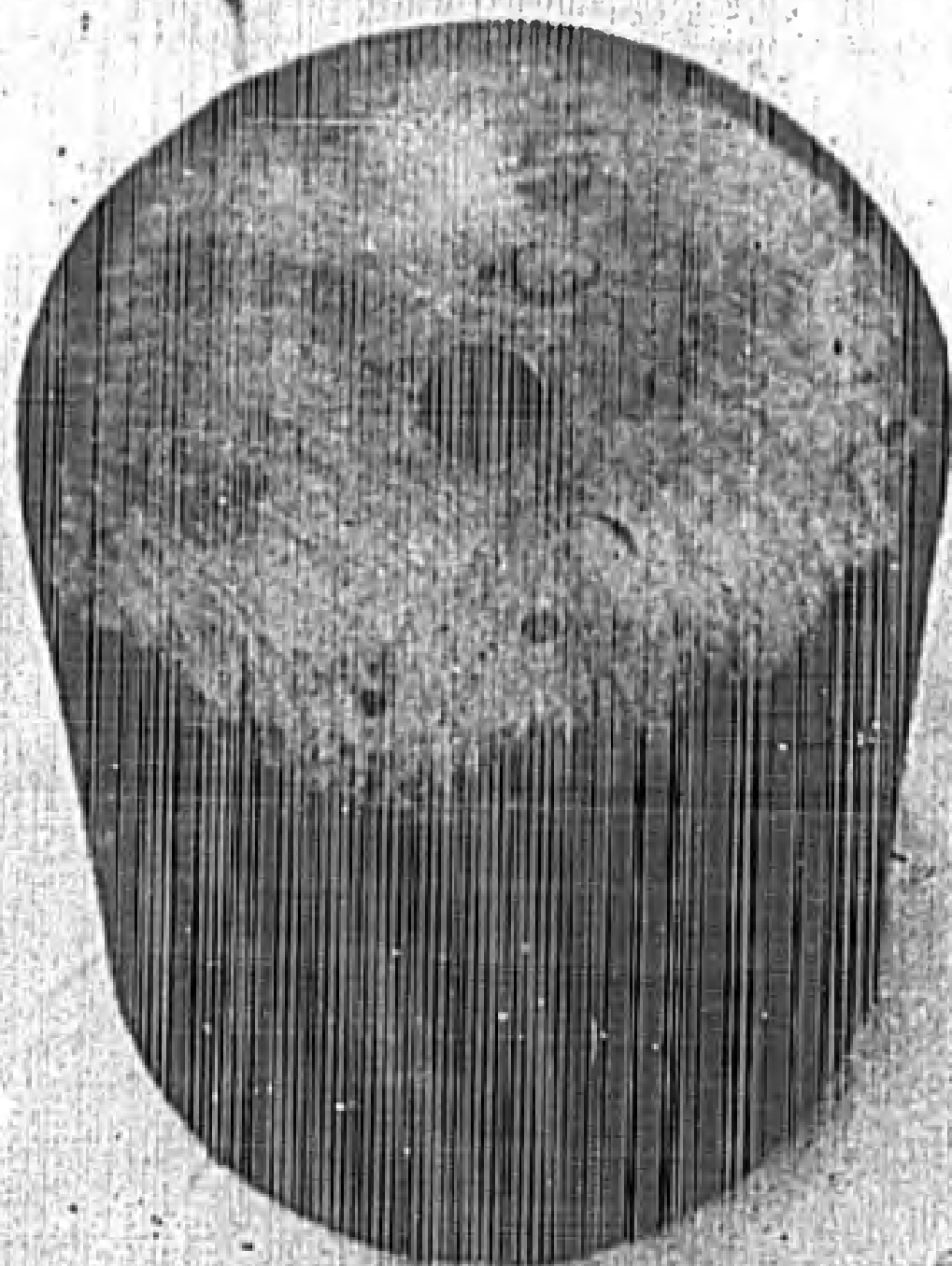


Figure 4. Trauzl Block With Detonator and Sample Holder

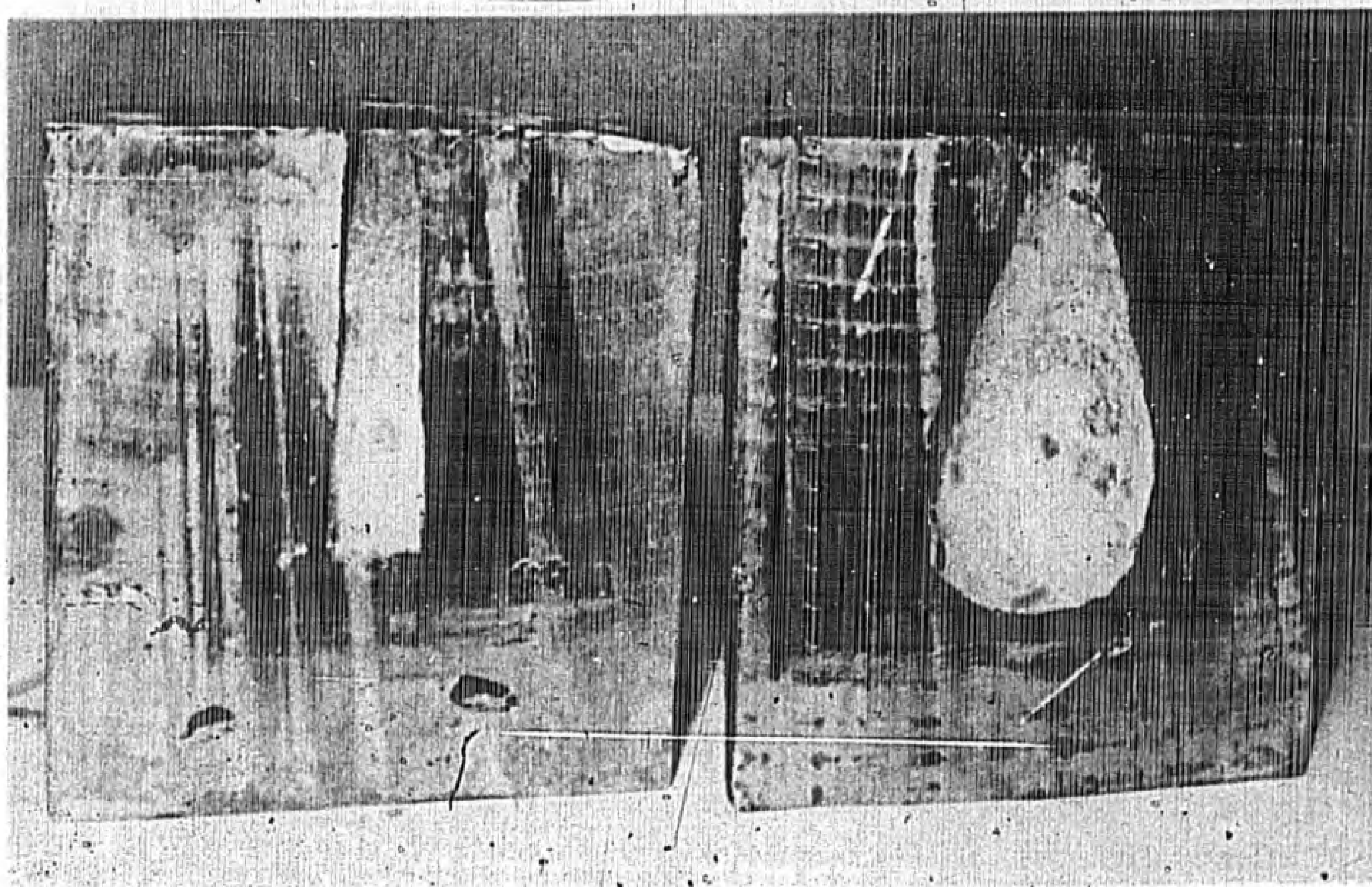


Figure 5. Trauzl Block Sections

TABLE VI. TRAUZL TESTS OF ELECTROLYTE

Test No.	Charge Composition (10 g)	Experimental Trauzl Expansion (cm ³)	Published Trauzl Expansion (cm ³)
1	Detonator + 10 ml H ₂ O	10	—
2	Nitromethane	332	352
3	2M LiClO ₄ - Nitromethane	358	—
4	2M LiClO ₄ - 80% Nitromethane - 20% Ethylene Carbonate	337	—
5	2M LiClO ₄ - 80% Nitromethane - 20% Propylene Carbonate	300	—
6	0.5M LiClO ₄ - 80% Nitromethane - 20% Propylene Carbonate	226.5	—
7	TNT	—	285

of "hot spots" in the sample at impact), with the grit side facing the sample, again without reaction.

In an effort to increase the sensitivity of the test, a holder was made which had a cylindrical 1/4-inch-diameter cavity with a sliding piston to fit. The sample was placed in the cavity, the piston put in place, and the plunger held off the piston by a spring. The dropping weight drove the plunger and the piston against the sample in the cavity. Again, there was no evidence of sample reaction when the 2-kg weight was dropped from 150 cm. Some of the liquid sample was driven past the cylinder and out of the cavity on this test, and liquid was also forced out of the filter paper during the drop on the original test. Some sample was also lost by evaporation, but the sample was larger (0.5 ml) than is commonly used (0.2 ml).

Within the limitations of the test equipment, the electrolytes prepared were no more sensitive than nitromethane, which has a sensitivity similar to that of TNT.

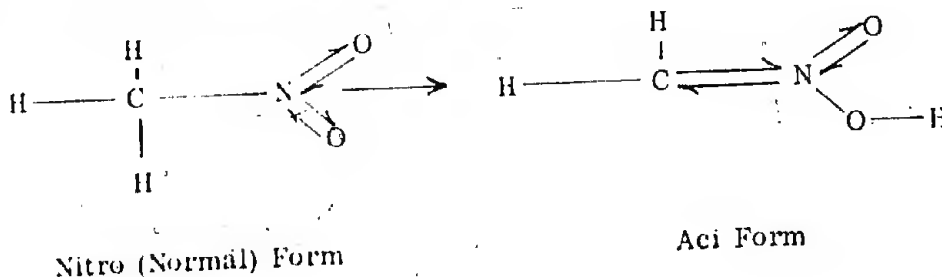
3.1.5 Electrolyte Stability

Material Compatibility. Materials that might be used as separators or for construction in contact with electrolyte were tested for corrosion resistance to a typical

electrolyte. For the test, 5 ml of electrolyte consisting of 2M LiClO_4 -80 percent nitromethane-20 percent propylene carbonate were placed in glass vials and samples of material were partially submerged in the liquid. The vials were sealed with Teflon[®] -lined caps and stored in an inert atmosphere box. The samples were examined visually for color changes, dissolution, or attack, with the results summarized in Table VII. Discoloration in the case of the samples that contained metal was probably electrolyte decomposition; in the case of the organic materials, it was color leaching or material attack.

Cell Stability and Electrical Tests. In the initial stages of this contract, an electrolyte of 2M LiClO_4 in 80 percent nitromethane-20 percent propylene carbonate was selected on the basis of physical properties and electrical conductivity. Cells were prepared with lithium anodes and cupric fluoride-graphite cathodes which operated at a reasonable current density but had a short life. Compatibility tests indicated that the anhydrous cupric fluoride was slightly soluble in the electrolyte, as indicated by a green coloration, but that the basic fluoride (CuOHF) was insoluble. The latter salt is precipitated from a boiling solution of $\text{CuF}_2 \cdot \text{H}_2\text{O}$ in water. Cells were prepared with CuOHF -graphite cathodes, lithium anodes, and 2M LiClO_4 -80 percent nitromethane-20 percent propylene carbonate electrolyte in plastic bags. When the cells were filled with electrolyte, heat developed and, in one case, the electrolyte ignited and burned in the argon atmosphere glove box.

Nitromethane exists as two tautomeric forms, as shown in the sketches below, with the nitro form usually predominating and thermodynamically the more stable.



The aci form is considered to be detonator sensitive, and a number of chemicals that make nitromethane more detonator sensitive are believed to function by moving the equilibrium toward the aci form. Conversely, some additives move the equilibrium toward the more stable nitro form. Basic materials generally shift the equilibrium toward the aci form, and the active hydrogen of the aci form might be expected to react with lithium.

In the case of the reaction in the assembled cell, the following possibilities were considered:

- Nitromethane reacts directly with lithium. (Nitromethane containing electrolytes of salts other than LiClO_4 has been used in lithium anode cells without reaction.)
- The LiClO_4 contains a reactive impurity.
- Reaction products on the lithium surface such as lithium nitride or lithium hydroxide shift the nitromethane equilibrium toward the aci form, which then reacts with the lithium.

TABLE VII. STABILITY OF MATERIALS IN ELECTROLYTE MATERIAL

Material	After 16 Hours	After 23 Days	After 78 Days
Aluminum Alclad® 2024-T-36	—	—	—
Stainless Steel No. 301	—	—	—
Magnesium AZ31B-H24	—	Slight Pitting	Pitting and discolora- tion
Stainless Steel/Brass, Soft Soldered	—	Discoloration	Discoloration
Stainless Steel/Brass, Hard Soldered	—	—	Discoloration; sediment
Zinc 3-9's Pure	—	—	—
Dacron® Nonwoven Fabric 3M-7020	—	—	—
Dacron® Nonwoven Fabric KM EM341	—	—	—
Dynel® Nonwoven Fabric KM EM341	Dissolved	Dissolved	Dissolved
Nylon Nonwoven Fabric NFM 100F1	—	—	Slight dis- coloration
-Polypropylene Nonwoven Fabric PE T12891	—	—	Slight dis- ation
Polyurethane Sponge	—	Jelled	Jelled
O-Ring Neoprene	—	—	Discoloration
O-Ring Ethylene Propylene Rubber	—	—	—
O-Ring Viton	—	—	Discoloration
O-Ring Buna-N	—	—	Discoloration
Fiberglas® -Epoxy G-10	—	Attacked	Delamination and attacked
Cast Epoxy Scotchcast No. 504	—	—	—
Acrylic Sheet Lucite®	Attacked	Attacked	Attacked
Microporous Rubber	Discoloration	Discoloration	Discoloration
Teflon® FEP	—	—	—
Teflon® TFE	—	—	—
Polyethylene	—	—	—
Polypropylene	—	—	—
Cellulosic Nonwoven Fabric KM R2401	—	—	—
3M: Minnesota Mining & Manufacturing KM: Kendall Mills NFM: National Filter Media PE: Pellon Company			

- Water in the system (either directly or after reaction with lithium to form LiOH) causes the nitromethane equilibrium to shift toward the reactive form.
- LiClO₄ causes the nitromethane equilibrium to shift toward the reactive form.

In experiments to explore these possibilities, the following results were obtained:

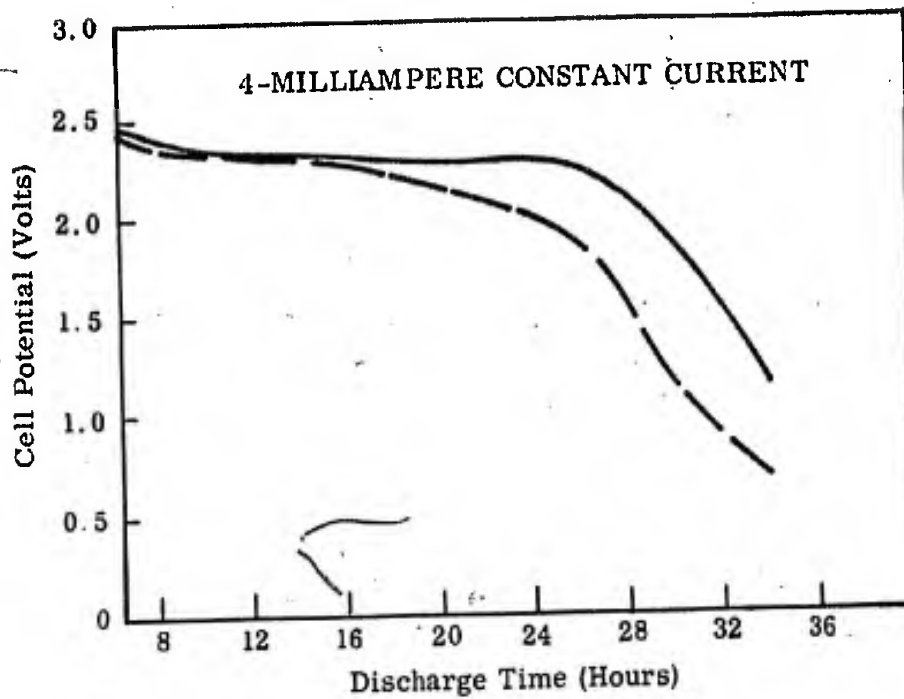
- Lithium shows no sign of reaction when immersed in nitromethane.
- No change of pH \pm 0.01 was evident when LiClO₄ was added to neutral water.
- Lithium etched in methanol with rinses in propylene carbonate and ethyl ether still reacted with the electrolyte. (The etch resulted in a bright metal surface but a very light tarnish returned during the rinsing.)
- Mixed nitromethane-propylene carbonate solvent contained 100 ppm of H₂O which increased to 150 ppm when the LiClO₄ was added.

In other compatibility tests, electrolytes containing ethylene carbonate rather than propylene carbonate appeared to be less reactive, possibly because the structure of this compound more closely resembles that of polycarbonate resin, which has been shown to have a stabilizing effect on nitromethane. Similarly, the addition of aluminum chloride decreased the reaction. Aluminum chloride was added as a dehydrating agent, but it may function as an acidic ion in the electrolyte to stabilize the normal form of the nitromethane. An electrolyte of 80 percent nitromethane-20 percent ethylene carbonate-2M LiClO₄-0.1M AlCl₃ was sufficiently nonreactive to be used for additional testing and was used for subsequent electrical and explosive tests.

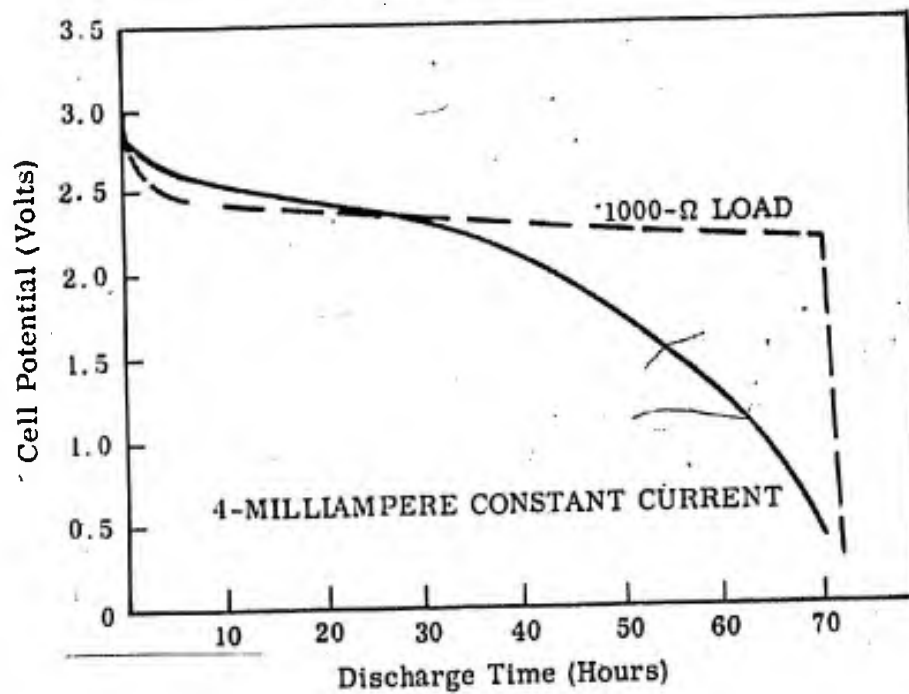
Two groups of cells were prepared in bags of laminated polyethylene, aluminum foil, and Mylar[®]. In assembling these cells, the cathode pellet with a copper grid was placed in a bag made of nonwoven polypropylene fabric. Two layers of glass paper were used to separate the cathode from a sheet lithium anode and nickel anode grid. Electrolyte was added and the trilaminate bag was heat sealed over grid tabs which served as connectors to the active electrodes. The results of these tests are shown in Figure 6. The active cathode area was 1.75 square inches, and the 4-milliampere discharge corresponds to a current density of 2.3 milliamperes per square inch. The abrupt termination of discharge of the cell (Figure 6b) after a 1000-ohm discharge was probably the result of shorting of the electrodes through the aluminum foil of the laminated bag.

3.2 CATHODE COMPOSITION

Prior to this effort, cupric fluoride cathodes were prepared which operated successfully with lithium anodes in cells for long discharge periods. The basic approach to eliminate water, which has a deleterious effect over long stand or discharge periods, was to prepare the electrode from copper carbonate and subsequently convert to copper salt to anhydrous cupric fluoride in a hydrogen fluoride atmosphere at 300°C. Reacting with hydrogen fluoride at elevated temperatures converts the salt to CuF₂ without conversion to any of the basic fluorides that form when CuF₂·2H₂O is heated in a normal atmosphere. Use of this technique simplifies electrode fabrication, since all the forming steps can be carried out in room atmosphere and only the final treatment and assembly require special atmosphere.



a. Cells 011301 and 011302



b. Cells 011101 and 011102

Figure 6. Discharge Voltage of Bag Cells

The solubility of CuF_2 in the electrolyte and the insolubility of CuOHF in the electrolyte made the latter material more desirable as the active cathode material. As previously mentioned, CuOHF is prepared by precipitating from a boiling solution of CuF_2 in water. The material was then washed, ball milled, and sieved through 40-mesh screen before being mixed with graphite to make the cathode mix. The CuOHF was mixed with the graphite in a twin-shell blender. No effort was made to optimize the grade of graphite or the amount used; 20 percent was chosen in the preparation of all cathodes used in cells.

3.3 ANODE FABRICATION AND SEPARATOR SELECTION

Lithium in extruded strip form (commercially available) was used without additional preparation in the cells constructed. The lithium is most conveniently cut with a molybdenum cutting wheel or with a plastic die.

Separators were not optimized in these tests, but glass paper was chosen for inertness and nonwoven polypropylene for its ease of heat sealing. After cutting to size, the material was used without other preparation.

SECTION IV CELL DESIGN

4.1 DESIGN PHILOSOPHY

4.1.1 Material Handling

The materials to be used in the assembly of nonaqueous electrolyte batteries are preferably handled in an inert atmosphere to prevent water pickup from the atmosphere or oxidation from the air. When lithium is used for the anode, argon must be used as the inert gas because lithium reacts with nitrogen as well as oxygen and water vapor. Material being passed into the glove box through the entry chambers carries some adsorbed water, so some provision must be made to remove water vapor from the atmosphere of the box. In addition, it has been found that a discernible amount of water vapor is in the as-received argon gas used as the chamber atmosphere. The argon used in the system is passed over titanium sponge at 600°C, at which temperature titanium reacts with oxygen, nitrogen, and water vapor. When the glove box is used, the atmosphere is continuously circulated through a cold trap to remove solvent from the atmosphere and then through a molecular sieve drying chamber. Part of the circulating gas can be shunted through a Beckman Hygromite meter to measure the water in the circulating gas - normally 7 to 10 ppm.

Operations such as soldering, inert arc welding, and metal rolling (Figure 7) can be performed in the glove box. Production cell assembly may advantageously be undertaken in the glove box, where the closely controlled atmosphere increases the reliability of the assembled product.

4.1.2 Cell Assembly

The models prepared for evaluation during this program did not have the necessary hardware for performance of all the functions considered, but the facilities for these functions should be considered in the design. Determination was made by the sponsor that the time spent in fabricating and testing the reservoir and activation system for this reserve battery could better be used in deriving electrochemical data and electrical performance characteristics of the battery. However, because the reservoir and activation system are inherent in the concept of this battery, consideration was given to the method of activation, material, physical shape, assembly, and mating with the cell pack. The location and placement of auxiliary electrodes to be used for timing and programming were also considered (a timing electrode being included in the cell pack design), as were electrodes for attitude and disturbance sensing. The method of fabrication of components and the assembly of critical parts such as the cell assembly and reservoir were given major consideration so that these parts that require a controlled atmosphere could be assembled in sealed units for subsequent line assembly of the completed unit. Mounting flanges or brackets and electrical connections were not included in the design at this stage since they will be entirely dependent upon the particular munition in which the battery unit is used.

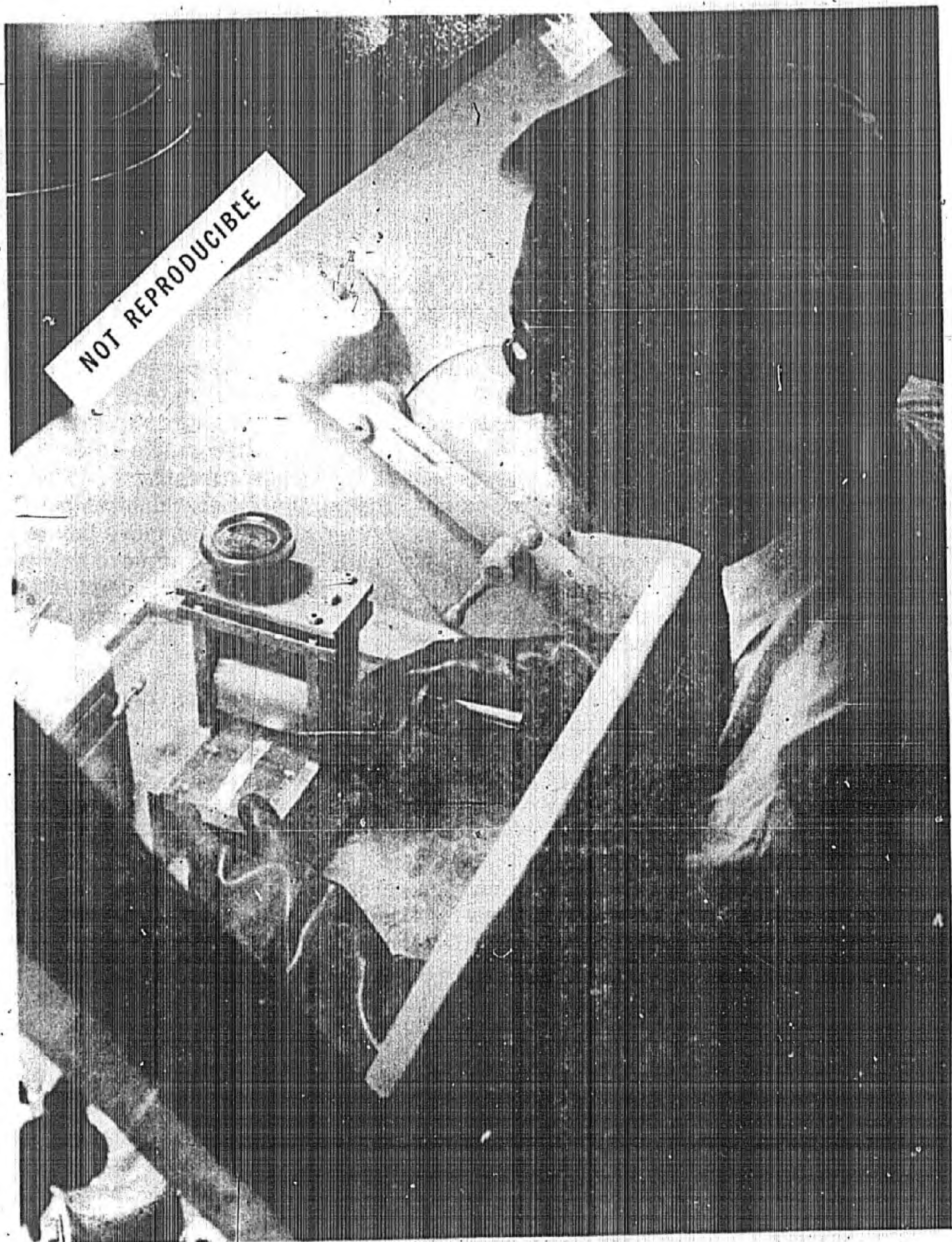


Figure 7. Rolling Lithium in an Inert Atmosphere Glove Box

A single cell should be sufficient to meet the voltage specification (three volts) of the electrical requirements for this program. A section of the designed battery with assembled cell pack and activator assembly is shown in Figure 8. The cylindrical configuration was chosen because it allows central feed during activation and also centers the detonator in the explosive electrolyte. The cylindrical shape has the additional advantages in the cell pack construction of allowing the use of circular electrodes and cell components that are susceptible to mechanized fabrication and of being readily stacked and placed during assembly.

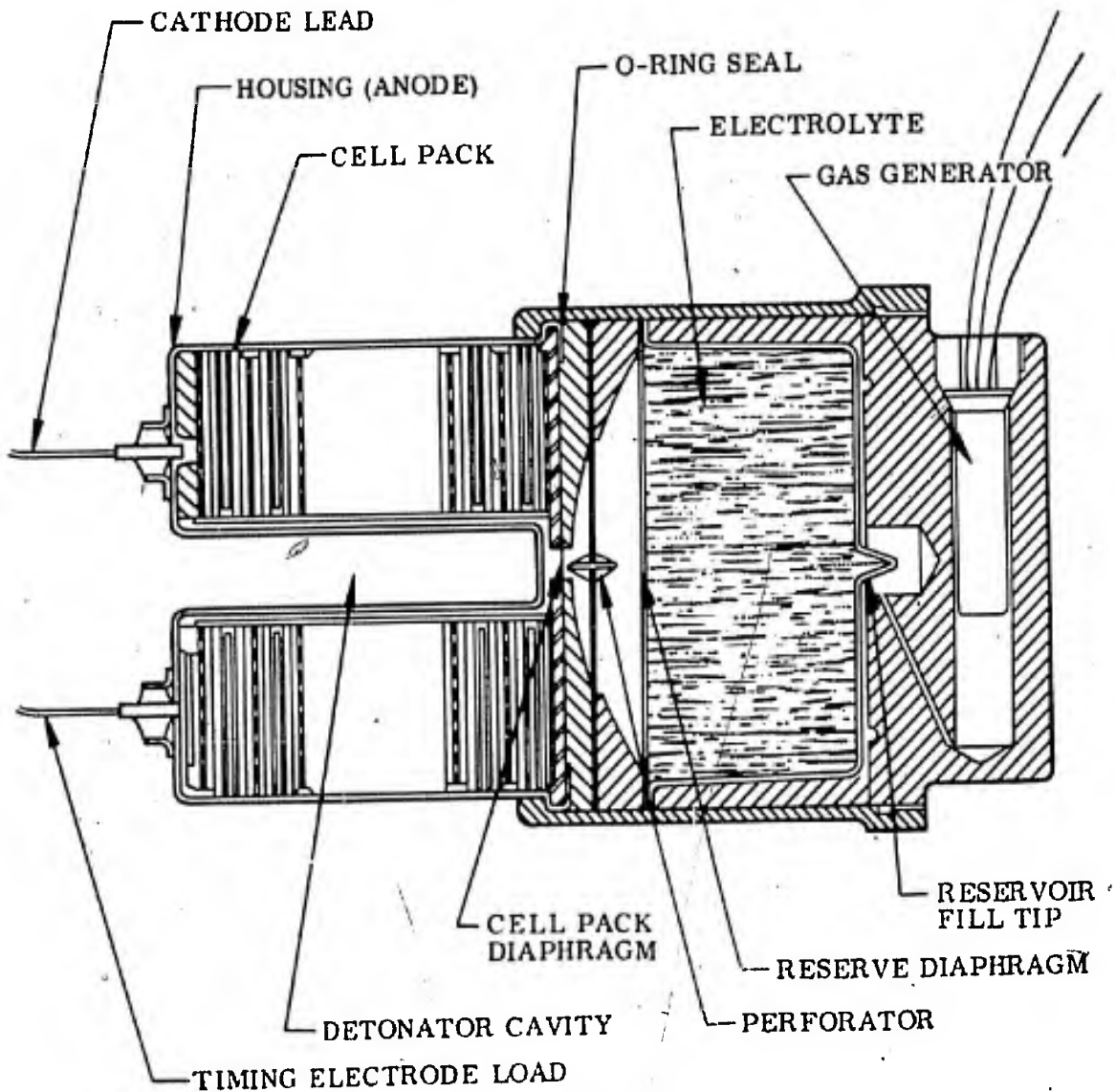


Figure 8. Reserve Battery Design

4.2 MECHANICAL DESIGN

4.2.1 Activator and Reservoir

Activation systems used for reserve-type batteries depend upon a pressure differential between the cell cavity and the reservoir at the time of activation to force electrolyte into the cell cavity. In most schemes, gas pressure is used as the driving force and, although gas stored under pressure and released by a squib valve can be used, pyrotechnic gas generators are now used almost universally.

For this program, a rolling diaphragm system was selected because it is compact, lightweight, symmetrical about an axis so that it can readily be mated to the cell pack, and lends itself to filling of the reservoir and sealing as a separate component of the final assembly. As pointed out earlier, it is advantageous to perform the filling in an anhydrous atmosphere rather than on the assembly line because in the latter environment the electrolyte is subject to water absorption. In addition, nitromethane has a vapor pressure at room temperature and limited toxicity that could trouble unprotected assemblers.

A gas generator was used to supply pressure. For the design used, an Eagle-Picher Model GG 156 with a 250-cc output was selected. This model was chosen for dimensional purposes; no extensive investigation of other sources was conducted. An expansion chamber was part of the generator housing to prevent high-pressure surges at initial activation. O-ring seals were used to prevent gas leakage outside the battery and past the reservoir diaphragm and electrolyte outside the battery. As can be seen in Figure 8, a threaded housing was used to attach the cell pack to the activator and also to assemble the reservoir and activator components. A hollow double-ended perforator was used to perforate both the reservoir and the cell pack diaphragms. Additional support for the reservoir diaphragm may be needed during shock before activation. The end use of the item and the mounting of the battery will dictate whether this is required.

4.2.2 Cell Pack

The electrodes for the cell pack were washer shaped (round with a round centrally located hole). One of these electrodes was placed on either side of a thin, perforated metal contactor which furnished electrical contact to the face of the electrode and also contact to the outside shell in the case of the anode, or to the internal contact tube in the case of the cathode. As can be seen in the detail of the cell pack in Figure 9, the anodes had a larger outside diameter and inside diameter than the cathodes, and insulator rings were used to prevent shorting of the electrodes to the counterelectrode current collector. The active electrode surfaces had an outside diameter of 1-7/8 inches and an inside diameter of 9/16 inches, with an area of 2.65 square inches on each face. If a conservative current density of one milliamperes per square inch is selected, four double-faced electrodes are needed for the 20-milliamperes current required.

Five unit cells with 10 anodes and 10 cathodes were used in the final cell design. Individual cathodes weighed 4.0 grams each and were 0.090 inch thick. Individual anodes were punched from a 0.020-inch-thick lithium strip. Separators consisting of

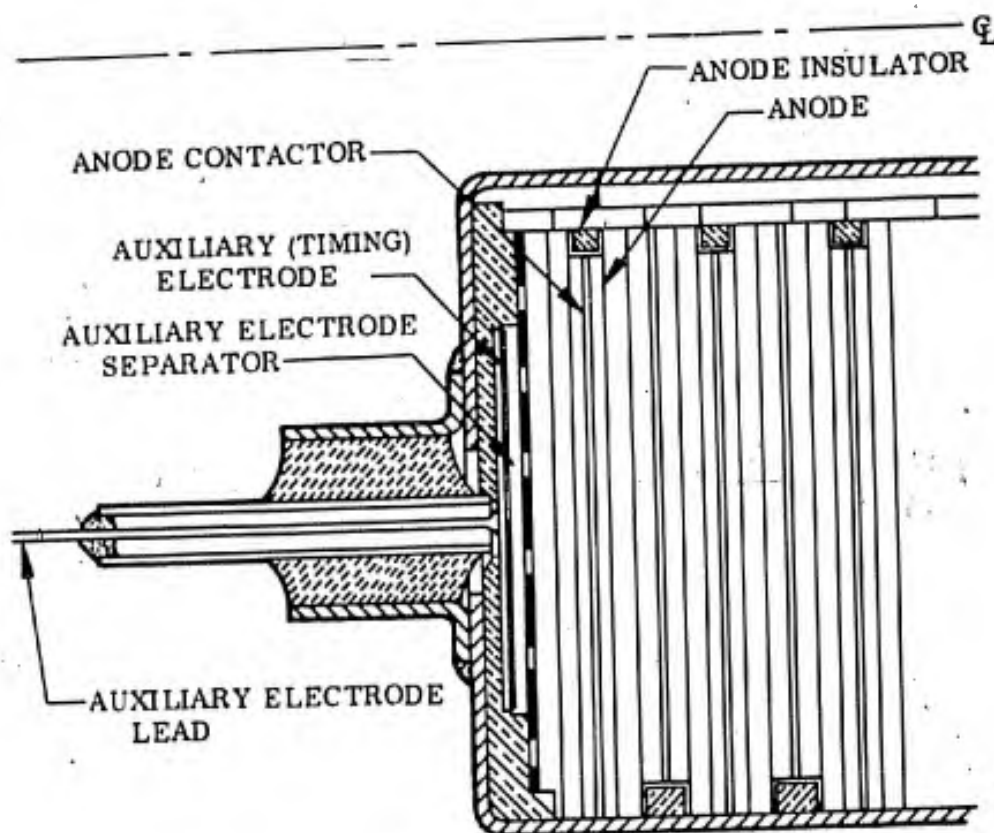
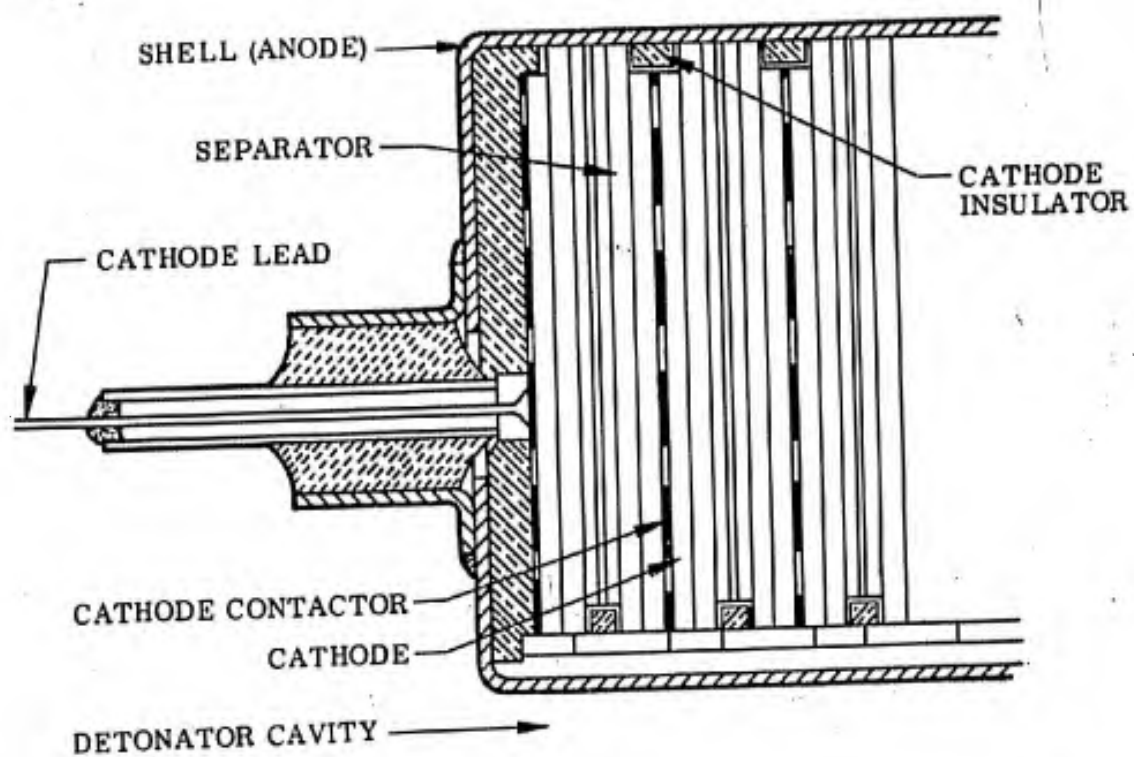


Figure 9. Cell Pack Detail

two layers of glass paper and two layers of nonwoven polypropylene fabric had a total thickness of 0.025 inch. The nickel anode collecting grids and the copper cathode collecting grids were of expanded metal pressed to 0.005-inch thickness.

Glass metal feedthrough terminals are shown in the design. In the cells that were assembled, an epoxy resin seal was made between the can and the cathode lead. The cathode lead itself was welded directly to the central cathode connecting tube so that the total battery current was not carried through contact pressure contacts. The parts used in the assembled cells are shown in Figure 10. The cover of the can was of copper-coated epoxy, which was soldered to the can for the final seal. Electrolyte was added through a tapped hole in the cover.

4.3 FABRICATION DIES AND FIXTURES

In the cell design, contact to the anode and cathode was made through electrode contactors which made point-multiple-point contact to the can or the central cathode tube for parallel connection of the electrodes. A controlled interference fit between the expanded metal contactors and their common terminal was necessary, so punch and die sets were made for each contactor. The dies with their ejector plates are shown in Figure 11, along with the other dies used in making cell parts. The cathode pressing die is shown next to the die sets and has a stationary outside cylinder and central core with a moving punch which compresses the cathode punch to the final cathode shape against a stationary plate. This pressing die was made to fit a standard metallographic press, which was a convenient source of controlled hydraulic power.

The lithium anodes were punched with a Delrin punch against polyethylene sheet. A plastic rather than metal punch was used because of the ability of lithium to wet and weld to metal readily, particularly when a clean lithium surface is exposed by shearing.

A simple "cookie cutter" punch was used to cut separators against a cardboard backing. As shown in Figure 11, the punch had a sponge rubber insert which served to remove the cut separator from the die.

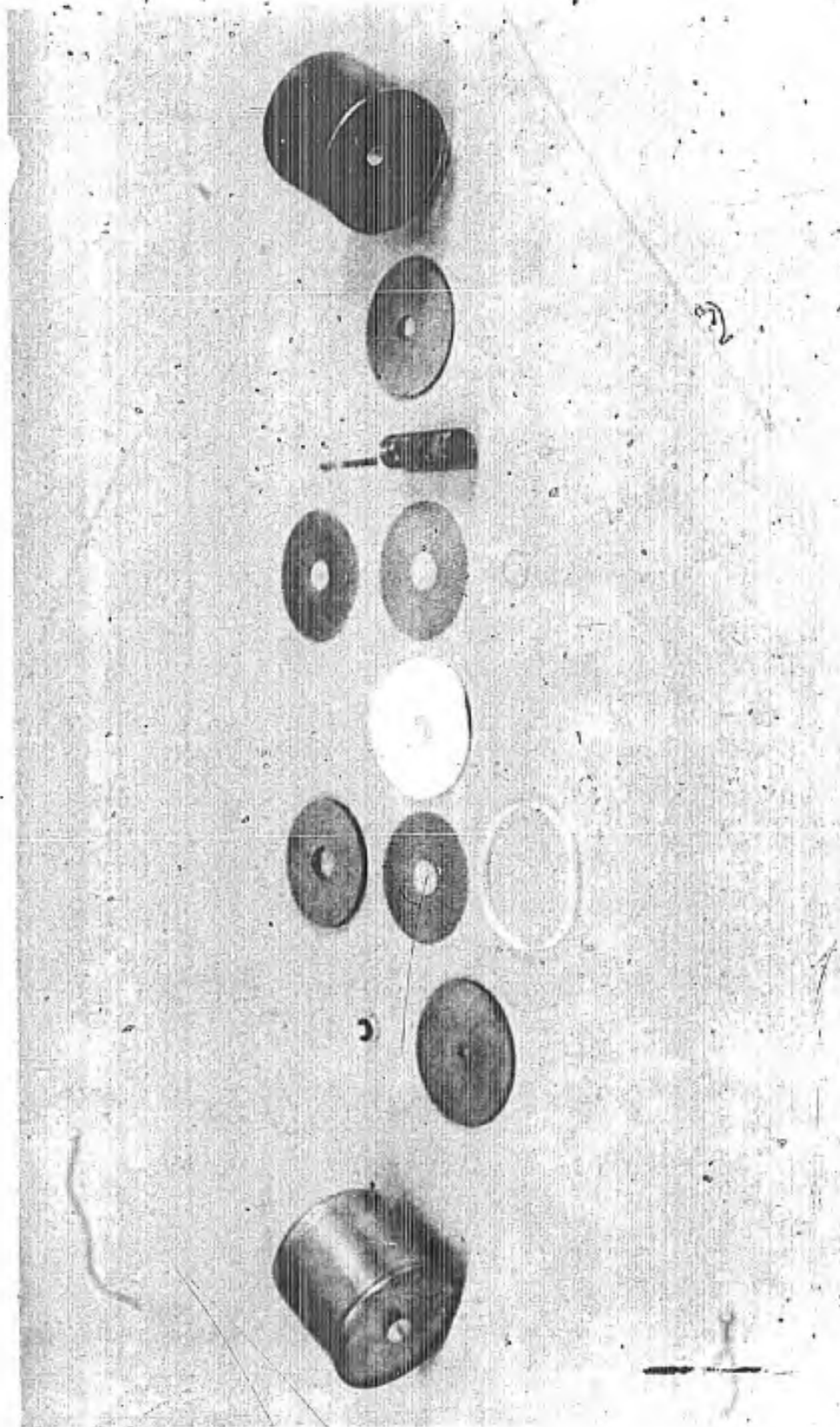


Figure 10. Cell Parts and Assembled Cell

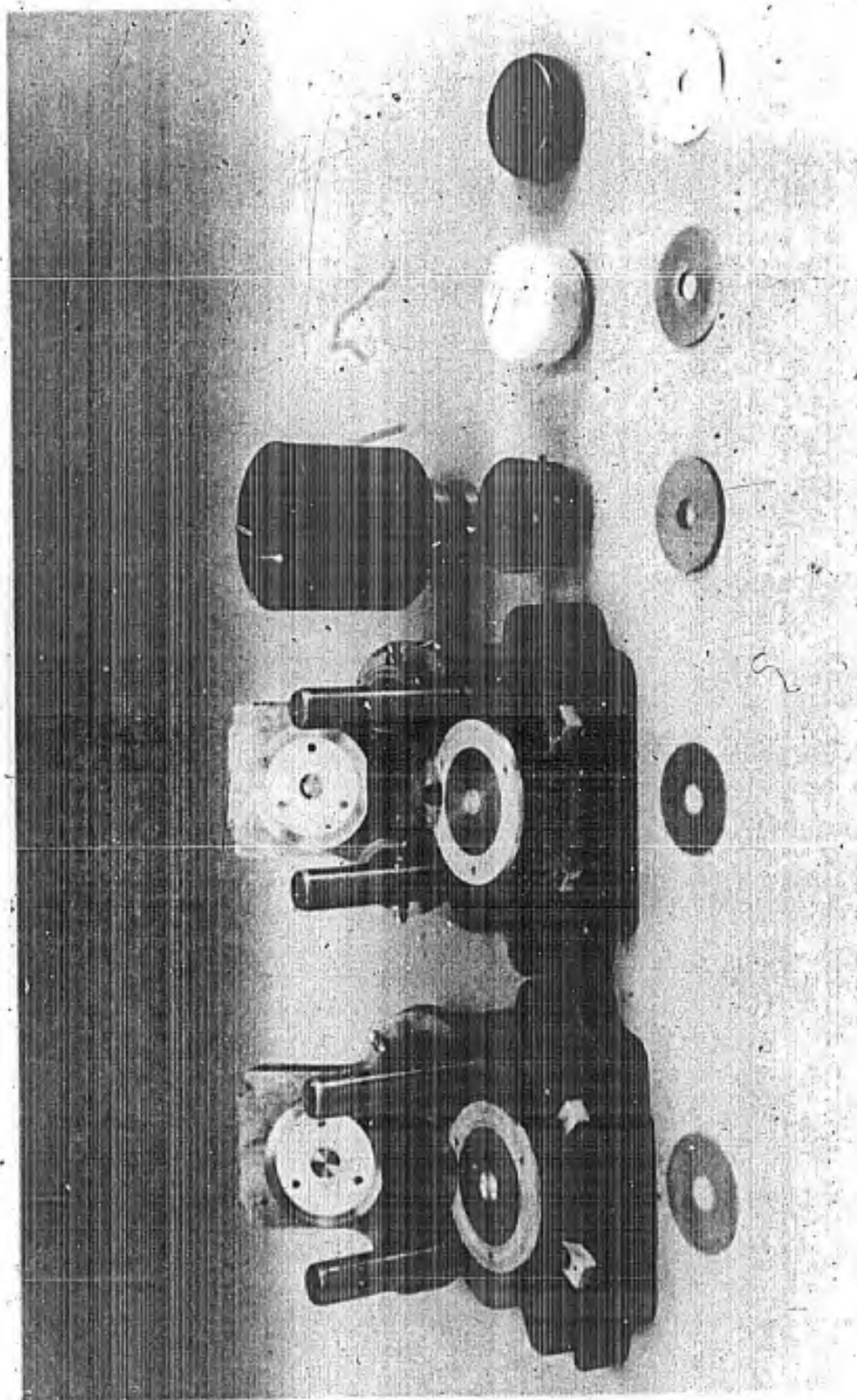


Figure 11. Cell Part Fabrication Punches

SECTION V CELL TESTS

5.1 ELECTRICAL DISCHARGE TESTS

Three cells were assembled in stainless steel cans, following the design outlined in paragraph 4.2.2. The first cell assembled (No. 3 cell on the explosion tests) was found to have a low-resistance short between terminals before electrolyte was added. This was subsequently found to be due to shrinkage of the bottom insulator in the can during cementing and curing, which in turn sheared material from the cathode during assembly and shorted to the can. The next cells assembled were resistance checked as each component was added. The cells as assembled had 27.5 square inches of apparent electrode surface. Cells No. 1 and 2 weighed 155 and 154 grams, respectively, and 44 and 45 grams of electrolyte was added to the cells. The electrolyte was 2M LiClO_4 -0.2M AlCl_3 -80 percent nitromethane-20 percent ethylene carbonate. The cells were given limited discharge (Figure 12) and removed for explosive test. The cell on 100-ohm load appears to be reaching a voltage plateau.

5.2 EXPLOSION TESTS ON CELLS

To determine if detonation would take place when the electrolyte was diluted with the cell components, two dummy cells were prepared in 2-inch-diameter stainless steel cans 1-7/8 inches high with a centrally located detonator cavity. The cell cans were filled with alternate layers of $\text{Cu}(\text{OH})_2 \cdot \text{CuCO}_3$ -graphite mix, expanded copper metal, more $\text{Cu}(\text{OH})_2 \cdot \text{CuCO}_3$ mix, a microporous rubber separator, a sheet of zinc (simulating lithium), and another separator, and these layers were repeated four times. The cell was closed with a 0.070-inch aluminum cover, epoxy cemented into place. The cells weighed about 120 grams dry after assembly. The control cell was filled with 50 grams of water and the other cell with 30 grams of 80 percent nitromethane-propylene carbonate-2M LiClO_4 , through a fill hole in the cover, and then sealed. The cells were fired separately with a No. 8 dynamite cap. The dummy cell was broken into three fragments (Figure 13) and metal components used in making the dummy electrodes were recovered and recognized. The cell with the nitromethane electrolyte had no recognizable fragments after detonation, except the can bottom. The 3/8-inch 6061-T651 aluminum plate on which it was exploded had a concave cavity about 1/2-inch deep at the point of explosion. In addition, the plate was deeply scarred in a radial pattern by the cell fragments.

The two cells previously discharged (Cells 1 and 2) and the cell with a short (Cell 3) were used for explosive tests. All cells were detonated with a No. 8 dynamite cap. Cell 3 was filled with electrolyte five minutes before detonation and, because of the electrical short, was quite warm when detonated. This cell had considerably greater effect on an aluminum witness plate on which the cells were fired than the other cells. Movies of this explosion were taken at 9,000 fps; however, the high light intensity

during the explosion washed out detail and fragment velocity could not be determined. The explosion flame had an immediate reddish cast which faded but later became a deeper red. Red is a characteristic color of lithium atom emission, and the colors during the explosion were presumably the result of LiClO_4 decomposition and lithium anode oxidation.

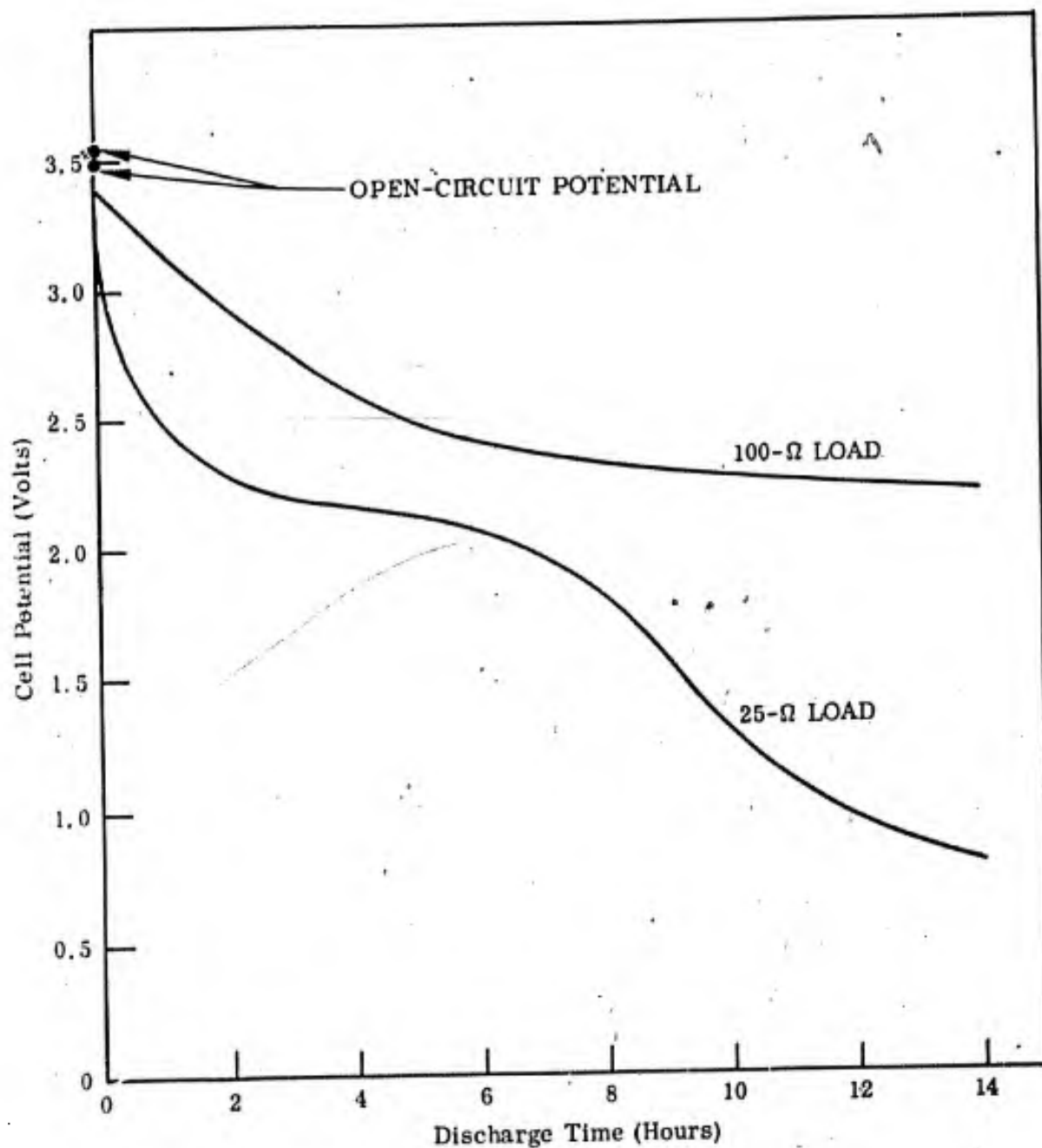


Figure 12. Discharge Voltage of Metal-Cased Cells

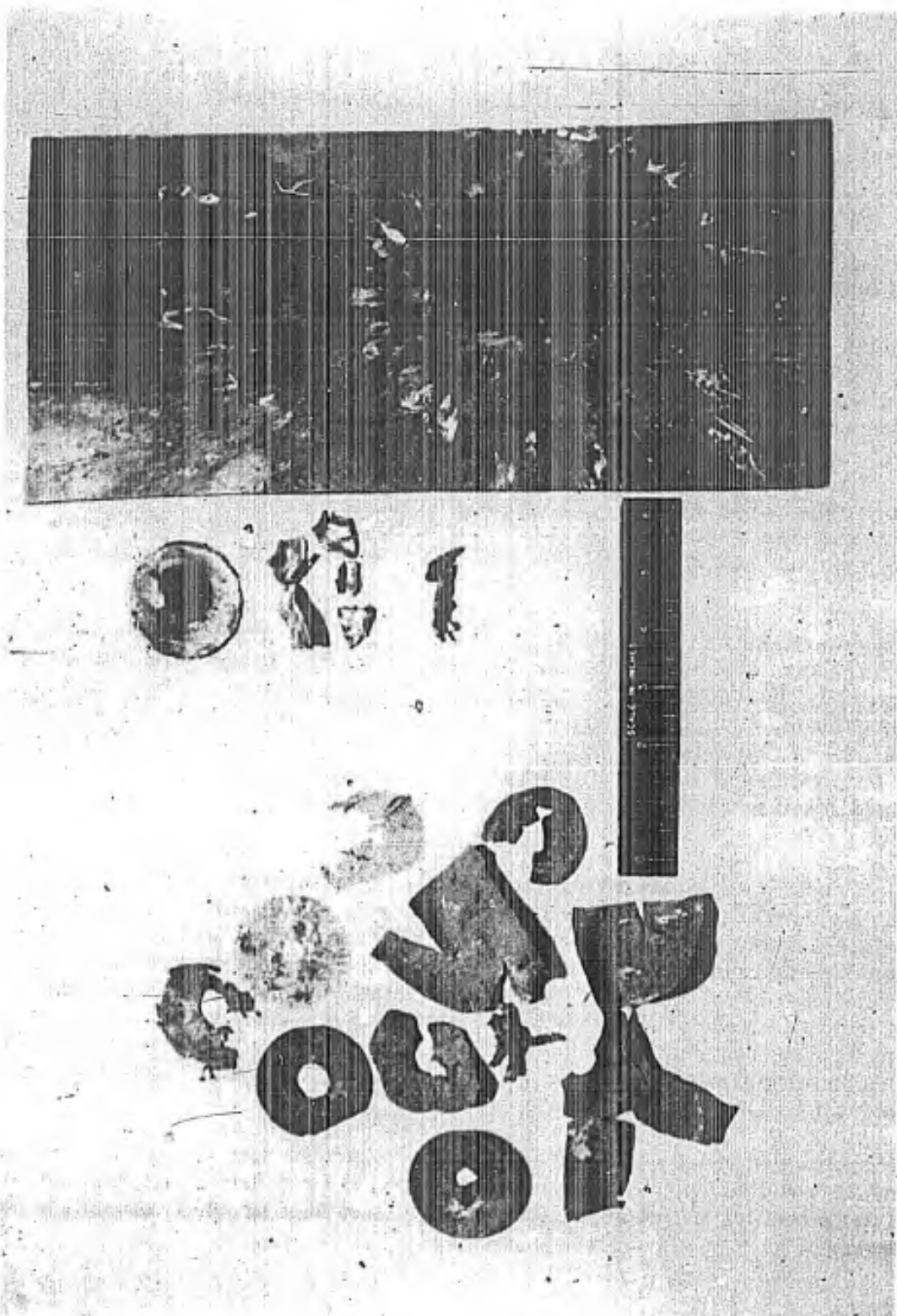


Figure 13. Explosion Test of Dummy Metal-Cased Cells With and Without Electrolyte

SECTION VI

TIMING, PROGRAMING, AND SENSING FUNCTIONS

Timing, programing, level indication, and disturbance sensing were studied as additional functions that could be included in the battery. Only limited experimental effort was made with these additional functions, but all appear feasible for inclusion in a final design. Timing is readily performed electrochemically; for a given amount of reactant, the time for the reaction to be completed is a function of the average current. These coulometric reactions are quite precise for a single reaction and are independent of temperature. In practice, a small area anode would be operated at low current density against one of the main cell cathodes at a constant current and the time of completion of the reaction signaled by an abrupt change in potential between the timing electrode and the cathode. The amount of active cathode material reacted by the timing electrode would be insignificant compared with that used by the power-producing reaction. A timing electrode of this type was included in the cell design of Figure 9.

A system similar in theory to that of the timing electrode was considered for programing functions. Instead of an anode of a single material, a layered electrode of two anode materials, both active but displaying different potentials when discharged against the power cathode, would be employed. The two layers would react at different potentials, the time of reaction depending again upon the average current between the electrodes. Mixed voltages may appear during the transition from one layer to the other, which would result in a less precise measurement than that with a single electrode material.

Level sensing would depend upon the resistance between combinations of insulated rod or strip electrodes partially immersed in electrolyte. A sampling of the resistance between different sets of electrodes would indicate if they were similarly immersed, which would be the case if the cell were level, or if they were immersed to different levels, in which case the increased resistance between certain electrode combinations would be indicative. Such a device could be incorporated in the present battery design by placing an insulating coating on the exterior of the detonator cavity and spacing film electrodes on this surface. Based on this approach, a simple laboratory device has been made which displays the inclination angle on a meter.

The same electrodes could be used for disturbance sensing except that the change in resistance would be sensed. Proper filtering and discrimination circuits could be included to account for harmonic motion resulting from wind or other naturally occurring phenomena.

SECTION VII

CONCLUSIONS

The major goal of this program, demonstration of the feasibility of a device that will supply electrical power and will also be capable of detonation, was achieved. Metal-cased cells were partially discharged and were then successfully detonated.

During the experimental work, it was found that the stability of lithium with the selected electrolyte was not sufficient for achievement of stand time and reliable operation. Insight into the reasons for the lack of stability was gained during the experimental work, and better stability was achieved by changes in the electrolyte. Approaches to take for further improvement were indicated by this work. The time consumed by this major problem reduced the amount of electrical testing that was undertaken, and no extended discharges were made with metal-cased cells.

A comprehensive effort to understand more completely the relationship between lithium, nitromethane, water, and ionizing salts soluble in the nitromethane should lead to the electrolyte stability necessary to complete the development of a useful multifunctional explosive battery.

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13. ABSTRACT The feasibility of a multifunctional explosive battery was established by the detonation of metal-cased cells which had previously been discharged to supply electrical power. These batteries were based on the lithium-copper hydroxyfluoride electrochemical system with an electrolyte of nitromethane, ethylene carbonate, lithium perchlorate, and aluminum chloride. The electrolyte was the explosive component in the cell and comprised less than 25 percent of the cell weight. A problem of limited compatibility exists between the lithium anode and the combination of nitromethane and lithium perchlorate in the electrolyte. Sufficient stability to complete testing was obtained by proper selection of electrolyte additives.			

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LINK B

LINK C

ROLE

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ROLE

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Multifunctional Explosive Battery
Metal-Cased Cells
Lithium-Copper Hydroxyfluoride
Electrochemical System
Nitromethane
Ethylene Carbonate
Lithium Perchlorate
Aluminum Chloride
Electrolyte Additives

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